It is believed that no fees are due in connection with the filing of this Preliminary Amendment. However, if any fees are inadvertently omitted or if any additional fees are required or have been overpaid, please appropriately charge or credit those fees to Conley, Rose & Tayon, P.C. Deposit Account Number 50-1505/5628-00403/EBM

Respectfully submitted,

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Strikethrough Version of Amended Claims

5. (Twice amended) The nanocomposite of claim ~~4~~ 1, wherein ~~any~~ the additional quaternary ammonium compound is a triester quaternary ammonium compound and wherein the triester quaternary ammonium compound comprises less than about 25 wt% of the quaternary onium compound mixture.

6. (Twice amended) The nanocomposite of claim ~~5~~ 1, wherein the fatty acids corresponding to the esters in the diester quaternary ammonium compound and the additional quaternary ammonium compound ~~quaternary onium compound mixture~~ have a degree of unsaturation such that the iodine value ~~("IV")~~ is from about 20 to about 90.

7. (Twice amended) The nanocomposite of claim ~~6~~ 1, wherein the additional quaternary ammonium compound is a triester quaternary ammonium compound, and wherein the diester quaternary ammonium compound ~~content is~~ comprises greater than about 60 wt% of the quaternary onium mixture, the triester quaternary ammonium compound ~~content is~~ comprises less than about 20 wt% of the quaternary onium mixture, and ~~the IV~~ wherein the fatty acids corresponding to the esters in the diester quaternary ammonium compound and the additional quaternary ammonium compound have a degree of unsaturation such that the iodine value is from about 30 to about 70.

8. (Twice amended) The nanocomposite of claim ~~7~~ 1, wherein the additional quaternary ammonium compound is a triester quaternary ammonium compound, and wherein the diester quaternary ammonium compound ~~content is~~ comprises greater than about 62 wt% of the quaternary onium mixture, the triester quaternary ammonium compound ~~content is~~ comprises less than about 17 wt% of the quaternary onium mixture, and ~~the IV~~ wherein the fatty acids corresponding to the esters in the diester quaternary ammonium compound and the additional

quaternary ammonium compound have a degree of unsaturation such that the iodine value is from about 40 to about 60.

9. (Amended) The nanocomposite of claim ~~8~~ 1, ~~wherein the IV~~ wherein the additional quaternary ammonium compound is a triester quaternary ammonium compound, and wherein the diester quaternary ammonium compound comprises greater than about 62 wt% of the quaternary onium mixture, the triester quaternary ammonium compound comprises less than about 17 wt% of the quaternary onium mixture, and wherein the fatty acids corresponding to the esters in the diester quaternary ammonium compound and the additional quaternary ammonium compound have a degree of unsaturation such that the iodine value is from about 45 to about 58.

13. (amended) The organoclay composition of claim 12, wherein the additional quaternary ammonium compound is a triester quaternary ammonium compound ~~is present~~ and wherein the triester quaternary ammonium compound comprises less than about 25 wt% of the quaternary onium compound mixture.

15. (Three times amended) The organoclay composition of claim 10, wherein the fatty acids corresponding to the esters in the diester quaternary ammonium compound and the additional quaternary ammonium compound ~~quaternary onium compound mixture for the quaternary ammonium compounds~~ have a degree of unsaturation such that the iodine value ~~("IV")~~ is from about 20 to about 90.

16. (Twice amended) The organoclay composition of claim ~~15~~ 10, wherein the additional quaternary ammonium compound is a triester quaternary ammonium compound, and wherein the diester quaternary ammonium compound ~~content is~~ comprises greater than about 60 wt% of the quaternary onium mixture, the triester quaternary ammonium compound ~~content is~~ comprises less than about 20 wt% of the quaternary onium mixture, and ~~the IV~~ wherein the fatty acids

corresponding to the esters in the diester quaternary ammonium compound and the additional quaternary ammonium c mpound have a degree of unsaturation such that the iodine value is from about 30 to about 70.

17. (Twice amended) The organoclay composition of claim ~~16~~ 10, wherein the additional quaternary ammonium compound is a triester quaternary ammonium compound, and wherein the diester quaternary ammonium compound ~~content is~~ comprises greater than about 62 wt% of the quaternary onium mixture, the triester quaternary ammonium compound ~~content is~~ comprises less than about 17 wt% of the quaternary onium mixture, and ~~the IV~~ wherein the fatty acids corresponding to the esters in the diester quaternary ammonium compound and the additional quaternary ammonium compound have a degree of unsaturation such that the iodine value is from about 40 to about 60.

18. (Amended) The organoclay composition of claim ~~17~~ 10, ~~wherein the IV~~ wherein the fatty acids corresponding to the esters in the diester quaternary ammonium compound and the additional quaternary ammonium compound have a degree of unsaturation such that the iodine value is from about 45 to about 58.

32. (Amended) The method of claim 31, wherein the ~~organoclay comprises a~~ diester quaternary ammonium compound is present as greater than 55 wt% of the quaternary onium compound mixture.

33. (Amended) The method of claim 31, wherein the additional quaternary ammonium compound is ~~organoclay comprises~~ a triester quaternary ammonium compound ~~present~~ and wherein the triester quaternary ammonium compound comprises less than about 25 wt% of the quaternary onium compound mixture.

quaternary ammonium compound and the additi nal quaternary ammonium compound have a degree of unsaturati n such that the iodine value is from about 45 to about 58.

ORGANOCLAY COMPOSITIONS PREPARED FROM ESTER QUATS AND COMPOSITES BASED ON THE COMPOSITIONS

FIELD OF THE INVENTION

This invention relates generally to organophilic clays (hereinafter referred to as "organoclays"), and more specifically relates to organoclays prepared from smectite clays which have been treated with a quaternary ammonium compound of a type commonly referred to as an ester quat. Such ester quats are derived from alkanolamine compounds whose hydroxyl groups are at least partially esterified with carboxylic acids to form a molecule with significant oleophilic properties. The resultant organoclays are useful as functional additives for organic based systems, where they may confer desired mechanical or physical properties sought for such systems.

BACKGROUND OF THE INVENTION

Organoclays represent the reaction product of a smectite-type clay with a higher alkyl containing ammonium compound (often a quaternary), and have long been known for use in gelling of organic liquids such as lubricating oils, linseed oil, toluene and the like and for use as rheological additives in a variety of organic based liquid systems and solvents. The general procedures and chemical reactions pursuant to which these organoclays are prepared are well known. Thus under appropriate conditions the organic compound which contains a cation will react by ion exchange with clays which contain a negative layer lattice and exchangeable cations to form the organoclay products. If the organic cation contains at least one alkyl group containing at least ten carbon atoms then the resultant organoclays will have the property of swelling in certain organic liquids. Among the further prior art patents, which discuss

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at length aspects of the preparation and properties of organoclays are U.S. Patents Nos. 2,531,427~~,~~; 2,966,506~~,~~; 3,974,125~~,~~; 3,537,994~~,~~; and 4,081,496~~.~~, all of which are incorporated herein by reference.

As utilized in the present specification, the term "smectite" or "smectite-type clays" refers to the general class of clay minerals with expanding crystal lattices, with the exception of vermiculite. This includes the dioctahedral smectites which ~~consist of~~include montmorillonite, beidellite, and nontronite, and the trioctahedral smectites, which includes saponite, hectorite, and sauconite. Also encompassed are smectite-clays prepared synthetically, e.g. by hydrothermal processes as disclosed in U.S. Patents Nos. 3,252,757; 3,586,468; 3,666,407; 3,671,190; 3,844,978; 3,844,979; 3,852,405; and 3,855,147, all of which are incorporated herein by reference.

In addition to their functions as thixotropes, organoclays find numerous other applications. Of particular interest for present purposes are composite materials composed of an organic polymer and a smectite-type clay mineral, with the mineral being ~~connected~~ coupled to the polymer through ionic or other bonding. Prior art pertinent to such composites include U.S. Patent No. 2,531,3963, incorporated herein by reference, published November 28, 1950, wherein a reinforced elastomer is disclosed. Smectite clays such as bentonite and hectorite are base exchanged with organic amines or salts thereof such as triethanolamine hydrochloride. Quaternary ammonium compounds can also be used. The resulting compounds, which are therefore "organoclays", are added to the lattices of elastomers. The organoclays can be added to the latex of any elastomer including natural rubber, and a large list of polymers and/or copolymers is provided. The resulting compositions can be vulcanized.

Japan Laid Open Application S51(76)-109998, deriving from application SHO 50(1975)-3580 was published Sept 29, 1976, and is entitled "Method for

Manufacturing a Clay-Polyamide Composite". It discloses a method for manufacturing a clay-polyamide composite characterized by carrying out the polymerization of lactam in the presence of ~~a clay-organic compound~~an organoclay ~~composite~~ made by carrying out ion exchange to bond an organic compound which contains at least one amino group and has the catalyst effect of polymerizing the lactam and clay. The organic compounds mentioned include omega-aminocapronic acid, a nylon salt, hexamethylenediamine, and aminodecanoic acid. The lactams include epsilon-caprolactam and others such as omega-enantolactam, omega-capryllactam, and omega-laurolactam. The clays used include the montmorillonite group of clay minerals such as montmorillonite, hectorite, etc; and other clays are listed. Montmorillonite is preferred because of the high exchange capacity. The composite is made by first ion exchanging the clay with the organic compound under aqueous conditions, after which the which the suspension is washed, filtered and dried, then crushed. (This is essentialiy the conventionai procedure for preparing an organoclay.) The "organoclay" and lactam are mixed, with the organoclay being 10 to 75 wt% of the mixture. During mixing the mixture is brought to 80-100 deg C to melt the lactam. Polymerization is carried out at 240 to 260 deg C. In the resulting composite product it is stated that the silicate layer has a thickness of 9.6 Angstroms. In a first example the interlayer distance of the organoclay layers before polymerization was 3.4 Angstroms, and 13.1 Angstroms after polymerization. In Example 4 the interlayer distance was 6.5 Angstroms before polymerization, and 50.6 Angstroms after polymerization. The composite produced is stated to have good fire-retardant properties, and improved mechanical properties.

Similarly, in ~~Kawasumi et al.,~~ U.S. Patent No. 4,810,734, which is incorporated herein by reference, a process is disclosed wherein a smectite-type clay mineral is contacted with a swelling agent in the presence of a dispersion medium thereby forming a complex. The complex containing the dispersion medium is mixed

Thus in a typical procedure for preparing a nanocomposite, the smectite clay, most commonly a montmorillonite, is treated with an organic ammonium ion to intercalate the organic molecule between the silicate layers of the clay, thereby substantially swelling or expanding the interlayer spacing of the smectite. (The reaction product resulting from this treatment may in accordance with the foregoing discussion, be referred to herein as an "organoclay"). Thereafter the expanded silicate layers are separated or exfoliated in the presence of or with the assistance of a polymer with which groups on the intercalated organic molecule are compatible. A monomer can also be used which is polymerized after being intermixed with the intercalated clay.

SUMMARY OF THE INVENTION

Now in accordance with the present invention, it has unexpectedly been discovered that organoclays based on specific types of ester quataternary ammonium compoundss, are remarkably effective for use in preparing nanocomposites. These organoclays comprise the reaction product of a smectite clay and a quaternary ammonium compound (hereinafter simply "quat") which comprises two esterified radicals (hereinafter called a "diester quat"). The diester quat may be present in admixture with further quaternary ammonium compounds having esterified radicals, especially compounds having three esterified radicals (hereinafter "triester quats"); or compounds having a single esterified radical (hereinafter "monoester quats"). Where such a mixture of quats is used, the reaction is between the smectite clay and the quat mixture. ~~The~~ In an embodiment, the diester quat should be present as greater than 55 wt% of the quaternary mixture; and the triester quat should be less than 25 wt%, with the fatty acids corresponding to the esters in the mixture having a degree of unsaturation such that the iodine value ("IV") is from about 20 to about 90. ~~More~~

~~preferably~~In an embodiment, ~~in such a mixture~~ the diester quat content is greater than 60 wt%, the triester quat content is less than 20 wt%, and the IV is from about 30 to about 70. ~~Yet more preferably~~In other embodiments, the diester quat content is greater than 62%, the triester quat content is less than 17 wt%, and the IV is from about 40 to about 60~~,~~. In some embodiments, an IV ~~and more optimally~~ from about 45 to about 58 may be desired.

BRIEF DESCRIPTION OF DRAWINGS

In the drawings appended hereto:

FIGURE 1 is a wide angle X-ray scan pattern for an organoclay in accordance with the present invention;

FIGURE 2 is a wide angle X-ray scan pattern for a clay-polymer nanocomposite prepared using the organoclay depicted in Figure 1;

FIGURE 3 is a wide angle X-ray scan pattern for a prior art ester quat-based organoclay;

FIGURE 4 is a wide angle X-ray scan pattern for a clay-polymer nanocomposite prepared using the organoclay the scan for which is depicted in Figure 2;

FIGURE 5 is a wide angle X-ray scan pattern for a prior art quat-based organoclay, where the quat does not include esterified radicals; and

FIGURE 6 is a wide angle X-ray scan pattern for a clay-polymer nanocomposite prepared using the organoclay the scan for which is depicted in Figure 5.

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DETAILED DESCRIPTION OF ~~THE INVENTION~~EMBODIMENTS

The quaternary ammonium compounds which are reacted with the smectite clays to produce the organoclays of the present invention are high in diester and low in triester content. They are obtained by reaction of C_{12} - C_{22} fatty acids or the hydrogenation products thereof, or a mixture of such acids, with an alkanolamine in the presence of an acid catalyst, wherein the ratio of fatty acid to alkanolamine is from about 1.40 to 2.0. The resultant ester amine reaction products are subsequently quaternized to obtain quaternary ammonium salts for reaction with the smectite. ~~The~~ In an embodiment, the fatty acid ~~is preferably~~may be a C_{16} - C_{22} acid containing a degree of unsaturation such that the iodine value ("IV") is in the range of from about 3-90, ~~preferably~~in other embodiments, from about 20-90, ~~more preferably~~in other embodiments, in the range of 40-60, and in ~~and still more preferably in~~ other embodiments in the ~~a~~ range of from about 45-55. ~~Preferred f~~Fatty acids include but are not limited to oleic, palmitic, erucic, eicosanic, and mixtures thereof. Soy, tallow, palm, palm kernel, rape seed, lard, mixtures thereof and the like are typical sources for fatty acid which ~~can~~ may be employed ~~in this aspect of the invention~~.

~~It is also preferred that~~ In an embodiment, the fatty acid(s) employed ~~in the present process~~may have a cis to trans isomer ratio of from about 80:20 to about 95:5. ~~More preferably~~In an embodiment, the trans isomer content of said fatty acid(s) is less than about 10%. ~~An optimum~~In an embodiment, the trans-isomer content is between about 0.5 - 9.9%. ~~The most preferred~~In an embodiment, the fatty acid is a mixture of tallow/distilled tallow having a cis:trans isomer ratio of greater than 9:1.

~~The~~ In an embodiment, alkanolamines ~~employable in the present invention generally correspond to~~may have the general formula:

R
|

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$$R_1 - N - R_2$$

wherein R, R_1 and R_2 are independently selected from C_2 - C_6 hydroxyalkyl groups. ~~Preferred a~~Examples of alkanolamines include but are not limited to triethanolamine, propanol diethanolamine, ethanol diisopropanolamine, triisopropanol amine, diethanolisopropanol amine, diethanolisobutanolamine and mixtures thereof.

~~The~~ In an embodiment, the molar ratio of ~~-~~fatty acid to alkanol ~~-~~amine is generally in the range of from about 1.4 to 2.0, ~~preferably~~ in other embodiments from about 1.55 – 1.90, in other~~, and more preferably, in~~ embodiments~~the range of~~ from about 1.65 – 1.75 and in~~. Best results are usually obtained when~~ other embodiments from ~~the molar ratio is between~~ about 1.68 – 1.72. The acid catalyst employable in ~~the present process~~an embodiment includes, but is not limited to, acid catalysts such as sulphonic acid, phosphorous acid, p-toluene sulphonic acid, methane sulphonic acid, oxalic acid, hypophosphorous acid ~~or~~ an ~~acceptable~~ Lewis acid in an amount of 500 – 3000 ppm based on the amount of fatty acid charge. ~~A preferred~~In an embodiment, an acid catalyst is hypophosphorous acid. ~~Typically~~In an embodiment, 0.02 – 0.2 % by weight, and ~~more preferably~~in other embodiments, 0.1 to 0.15 % by weight of acid catalyst, based on the weight of fatty acid, ~~are~~ may be employed ~~in the present process~~.

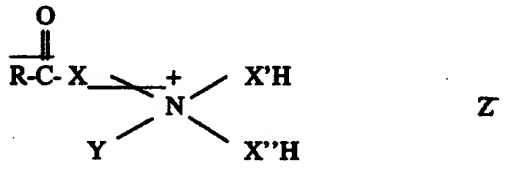
The esterification of fatty acids with alkanolamines is carried out at a temperature of from about 170° – 250°C until the reaction product has an acid value of below 5. After the esterification, the crude product is reacted with alkylating agents in order to obtain the quaternary ammonium product. ~~Preferred a~~Alkylating agents include C_1 – C_3 straight or branched chain alkyl halides, phosphates, carbonates, ~~-~~or sulfates, C_7 – C_{10} aralkyl halides, phosphates or sulfates, and mixtures thereof. Examples of ~~preferred~~ alkylating agents in an embodiment include, but are not limited to, methyl chloride, benzyl chloride, diethyl sulfate, dimethyl carbonate, trimethyl phosphate, dimethyl sulfate or mixtures thereof. Choosing the type and amount of

between 170°C to 250°C, is effective in minimizing triester formation in the ester amine mixture.

The quaternization may be carried out in bulk or in solvent, at temperatures ranging from 60° – 120°C. If a solvent is employed, then the starting materials and/or product ~~must~~ will, in many circumstances, be soluble in the solvent to the extent necessary for the reaction. Solvents of this type are generally known in the art. Suitable examples include polar solvents such as, for example, lower alcohols, ~~i.e.~~ e.g., C_1 – C_6 alcohols. Other solvents which ~~can~~ may be employed include, but are not limited to mono–, di–, and tri–glycerides, fatty acids, glycols and mixtures thereof.

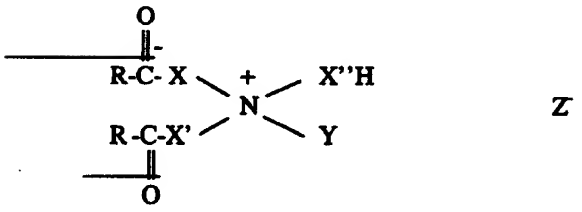
~~The preferred~~In an embodiment, the quaternary ammonium salt ~~for the invention comprises~~includes a mixture of mono - (I), di- (II) and triester (III) components of the following formulae:

I)



Z^-

II)



Z^-

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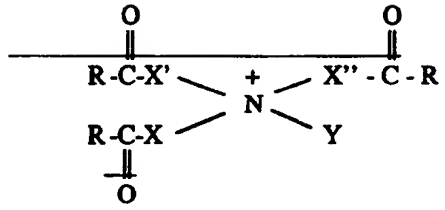
III)  Z^-

wherein:

X, X' and X'' are the same or different and are selected from straight or branched chain, optionally substituted oxyalkylene or polyoxyalkylene groups having from 2 to 6 carbon atoms, ~~preferably~~ in other embodiments, 3 to 6 carbon atoms, where the oxyalkylene units number from about 1 to 10, ~~preferably~~ in other embodiments, 1 to 5, and ~~still~~in ~~more preferably~~other embodiments, 1 to 2; each R group is individually selected from straight or branched chain, optionally substituted alkyl groups having from 11 to 23 carbon atoms, Y is an alkylphenyl group or a straight or branched chain optionally substituted C_1 to C_6 alkyl or alkylene group; and Z^- represents a softener compatible anion including, but not limited to, halogen, CH_3SO_4 or $C_2H_5SO_4$.

The reaction products may also ~~contain~~ include minor amounts of methyl trialkanolammonium salts and other impurities. The amount of diester in the final product (II) is generally greater than about 55% by weight and the amount of triester (III), based on the gas chromatograph of the ester amine, is generally less than about 25%, ~~preferably~~ in other embodiments, less than 20% by weight based on the total amount of quaternary ammonium salt product.

~~Typical~~ In an embodiment, product compositions contemplate an ester distribution within the following ranges: greater than about 55 wt% diester and less

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than about 25 wt% triester, with a total fatty acid IV of from about 20 to about 90; ~~more preferably~~in other embodiments, greater than about 60 wt% diester and less than about 20 wt% triester, with a total IV of from about 30 to about 70; and in still ~~more preferably~~other embodiments, greater than about 62 wt% diester and less than about 17 wt% triester, with a total IV of from about 40 to about 60. In many instances triester content will be in the 10.0 to 17.0 wt% range. In ~~a most preferred~~ an embodiment, the IV is between about 45 to about 58.

~~The~~ In an embodiment, the ratio of cis to trans double bonds of the above salts is ~~preferably~~ in the range of from about 80:20 to about 95:5. ~~Preferably~~In other embodiments, the cis:trans ratio is greater than about 90:10. In ~~a most preferred~~some embodiments, the amount of trans isomer ~~ideally~~ is in the range of from 5 to 9.5%.

There are several convenient methods for obtaining the desired cis:trans ratio of the quaternary ammonium salt product. ~~The preferred~~One method is to produce the quaternary ammonium salt from ~~a~~ cis-isomeric and trans-isomeric fatty acids after adjusting said acids to the desired ratio.

Another method is to produce the quaternary ammonium salt from the mixture after adjusting the ratio thereof by isomerizing a portion of the cis-isomeric fatty acid or ester thereof into the trans-isomer, in the presence of a metallic catalyst. Other methods are readily apparent to and well within the skill of one of ordinary skill in the art.

The quaternary ammonium compounds ~~according to the present invention~~ ~~can~~may generally be prepared by reacting at least one C_{12}-C_{22} fatty acid having a IV of from 20-90 with an alkanol amine in the presence of an acid catalyst. The ratio of acid to amine is ~~preferably~~, in one embodiment, in the range of 1.4 to 2.0, and the reaction is carried out at a temperature of from about 170°C to about 250°C until the

group is non-reactive and does not lead to the formation of a tri-substituted species. Accordingly, the heat up rate ~~which is an important requirement of the trialkanolamine based process~~ is less important when ether alkanolamines are employed as a reactant ~~since the formation of tri-substituted species is not possible.~~

Further, employment of ether alkanolamines is beneficial in that they are more reactive with a broader range of alkylating agents, and the final products are easier to formulate and are more storage stable. Finally, controlling the ratio of fatty acid/fatty acid methyl ester to ether alkanolamine may control the ratio of mono- and di-substituted species ~~can may be controlled by controlling the ratio of fatty acid/fatty acid methyl ester to ether alkanolamine~~.

~~A exemplary process~~In an embodiment, ~~for~~ the preparation of a high diester quaternary ammonium mixture ~~comprises~~ includes reacting:

I) a C_{11} - C_{23} substituted or unsubstituted fatty acid ~~or~~ mixture of fatty acids having an Iodine Value of from about 20 to about 90, and having less than about 20% trans double bonds, with

II) an ether alkanolamine of the formula:

$$R - N - R_1$$
$$|$$
$$R_2$$

wherein R is a C_2 - C_6 alkyl ether, and each of R_1 and R_2 is independently selected from C_2 - C_6 hydroxyalkyl groups, wherein the molar ratio of said fatty acid to ether alkanol amine is from about 1.4 to about 2.0, ~~preferably~~ in other embodiments, from about 1.6-1.9, and quaternizing the resultant ester amine mixture in order to obtain an improved high diester quaternary ammonium mixture.

~~Preferred e~~Ether alkanolamines ~~are selected from the group consisting of~~include, but are not limited to, methoxyethyldiethanolamine,

Nos. 4,664,842 and 5,110,501, both of which are incorporated herein by reference, (assigned to the assignee Southern Clay Products Inc.~~)~~) for further details of such mill. The conditions for use of the MG mill may, in ~~the present instance~~an embodiment, be substantially as in the said patents; e.g. the said pressure differential across the gap ~~is preferably~~may be in the range of from 70,300 to 562,400 g/cm^2 with 140,600 to 351,550 g/cm^2 being more typical in representative operations.

_____Depending upon the specifics of the equipment, pressures higher than 562,400 g/cm^2 ~~can~~ may readily be used. The slurry to be treated may be passed one or more times through the MG mill. Among additional instrumentalities which ~~can~~ may be effectively utilized ~~in the present invention~~ to provide high shearing of the clay component, is the rotor and stator arrangement described in the assignee Southern Clay Products' U.S. Patent No. 5,160,454, which is incorporated herein by reference. Following the high shear step, the slurry is intermixed with the quaternary ammonium salt and the reaction slurry ~~is preferably~~may again be subjected to high shearing by one or more passes through the MG or other mentioned instrumentalities. The slurry is thereupon dewatered, and the quaternary ammonium-treated clay dried and ground to provide a dry organoclay product.

_____When used in composites such as nanocomposites, the organoclay compositions of the invention yield unexpected improvements in the mechanical and other properties of the composite, including with respect to tensile strength, tensile modulus and flex modulus, all of which are highly significant attributes for the plastics and similar formulations.

_____The organoclays ~~of the invention can~~may be used in preparing nanocomposites by any of the methods which are set forth in the prior referenced patents, and with a large variety of polymerizable resins such as polyamides, epoxy, polyvinyl, polyacrylamide, etc.

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The invention will now be illustrated by examples, which are to be regarded as illustrative and not delimitative of the invention. Unless otherwise indicated to the contrary, all parts and percentages are by weight.

Example 1

An organoclay composition ~~in accordance with the invention~~ was prepared from a smectite mineral clay which was processed as above described, i.e. crushed, ground, slurried in water and screened, converted to its sodium form, and then subjected to high shear by being passed as a dilute slurry through an MG mill, and then as a slurry treated with the quaternary ammonium compound in accordance with the invention. This quaternary composition was a diester quat in admixture with further quaternary ammonium compounds having esterified radicals, especially compounds having three esterified radicals (hereinafter ~~"triester quats"~~); or compounds having a single esterified radical (hereinafter "monoester quats"). The reaction forming the organoclay was between the smectite clay and the quat mixture. The diester quat was present as greater than 55 wt% of the quaternary mixture; and the triester quat was present as less than 25 wt%, with the fatty acids corresponding to the esters in the mixture having a degree of unsaturation such that the iodine value ("IV") is from about 20 to about 90. A wide angle x-ray scan pattern for the product resulting from the reaction is shown in Figure 1, where the detected reflection intensity in counts/second is plotted against the D-spacing in Angstrom Units. The 001 reflection peak indicates a remarkably high D_{001} spacing for the organoclay of 59.1A, and suggests that the organoclay will exhibit a very high exfoliation efficiency in nanocomposites.

Example 2

_____5 wt% of the organoclay powder of Example 1 was premixed with high impact polystyrene ("HIPS") pellets by mechanical means. 50 to 60 g of this dry blend was added to a Brabender mixer which was then operated at 60 rpm. The temperature of the mixer was varied from 190° C to 230° C. The time of melt blending in the mixer was varied from 15 minutes to one hour. At the end of the prescribed time, the molten mixture was extruded from the Brabender. The resulting nanocomposite sample was prepared for x-ray analysis by pressing the mixture in a Wabash press with the platens heated to 150° C. at a pressure of 7,500 to 10,000 p.s.i. for one minute. A 1-1/8" by 1-1/8" square was cut from the sample for analysis. The resulting wide angle x-ray scan pattern is shown in Figure 2. The D_{001} reflection peak of the organoclay is completely gone in this composite indicating very high exfoliation of the organoclay in the HIPS matrix.

Example 3

_____In this Example an organoclay sample was prepared using the procedure of Example 1, except that in this instance the quat used was a diester quat (based on ~~hydrogenated~~ tallow), which in part differs from the quat used in Examples 1 and 2 in including methyl groups on the remaining two -N bonds, whereas the Example 1 quat includes a hydroxyethyl group on one of the said remaining -N bonds. A wide angle x-ray scan pattern for the product resulting from the reaction is shown in Figure 3 .The 001 reflection peak indicates a D_{001} spacing for the organoclay of 39.5A, which is not as high as the sample of Example 1, although still suggesting that the organoclay will exhibit a reasonably high exfoliation efficiency in nanocomposites.

Example 4

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4, indicates ~~a relatively inadequate~~less efficient exfoliation of the organoclay in the HIPS matrix.

While the present invention has been described in terms of specific embodiments thereof, it will be understood in view of the present disclosure, that numerous variations upon the invention are now enabled to those skilled in the art, which variations yet reside within the scope of the present teaching. Accordingly, the invention is to be broadly construed, and limited only by the scope and spirit of the claims now appended hereto.

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